DIONEX

PULSED ELECTROCHEMICAL DETECTOR
OPERATORS MANUAL

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Document No. 034177
Revision 01
September 1989

MP MASTER
MA32859

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PRINTING HISTORY

Revision 01 September 1989

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SECTION 1
INTRODUCTION

1.1 DIONEX PULSED ELECTROCHEMICAL DETECTOR

Congratulations on your purchase of a Dionex Pulsed Electrochemical Detector (PED). This detector will enable you to perform all applications using electrochemical detection. These include applications of conductivity detection, either suppressed or single column; DC amperometric applications such as the determination of phenols and catecholamines; and important new applications for carbohydrates, amines, and other species requiring integrated and pulsed amperometric detection. You can also use the detector for electroanalytical investigations using cyclic voltammetry.

The Pulsed Electrochemical Detector is operated either from the front panel, or from a computer via the parallel interface. In addition, limited control is available via TTL-level commands from a computing integrator. Detector output is available both as an analog signal for an integrator or strip chart recorder and as a digital output for computer acquisition of data by a Dionex computer and interface.

The remainder of Section 1 offers an introduction to PED detection modes and the detector cells. More detailed descriptions of these can be found in Section 3.

1.2 DETECTION MODES

1.2.1 CONDUCTIVITY

Conductivity detection is performed by applying a pulsed voltage to two electrodes in the conductivity cell. The measured electrical conductivity is dependent on the mobility of ions in the applied electric field; on their electrical charge; and most importantly, on their concentration. Unlike amperometric detection, no oxidation or reduction occurs during conductivity detection.

When the PED **MODE** is set to **CONDUCTIVITY**, the **ANALOG RANGE** and the detector output are in siemens (S).

The usefulness of conductivity detection is greatly enhanced by chemical suppression. The suppressor neutralizes ionic reagents added to the mobile phase. In addition, the electrical conductivities of the more strongly ionized analytes are enhanced when they are converted to the free acid or base forms. The most important advantage in using a suppressor is a large increase in signal-to-noise ratio.

1.2.2 AMPEROMETRY

Amperometric detection is based on the application of a constant DC potential between electrodes in the amperometric cell. The cell uses three electrodes. The electrode at which the analyte molecules are either oxidized or reduced is called the working electrode. In an oxidation, the molecules lose electrons to the electrode. Conversely, in a reduction, analyte molecules gain electrons donated by the electrode. Analyte concentration is determined by measuring the electric current resulting from the molecules gaining or losing electrons. If the analyte molecules are to be oxidized, the working electrode potential must be high enough to cause their oxidation, but not high enough to cause oxidation of interfering species in the sample or of components in the mobile phase. Since only species capable of being oxidized or reduced at the applied potential will be detected, amperometry is a highly selective and sensitive detection method. It is commonly used for easily oxidized species such as phenols, catecholamines, cyanide, and sulfite.

When the PED **MODE** is set to **AMPEROMETRY**, the **ANALOG RANGE** and the detector output are in amperes (A).

Cyclic voltammetry is performed in the **AMPEROMETRY** mode by applying a triangle wave to the amperometry cell.

1.2.3 INTEGRATED AND PULSED AMPEROMETRY

For several classes of molecules, amperometric detection can only be used if a repeating sequence of potentials is applied to the working electrode. These include carbohydrates (sugars); simple alcohols; species containing primary, secondary, or tertiary amines; and most species containing sulfur (with the exception of fully oxidized sulfur such as sulfates, sulfonates, and sulfones). For these species, both high and low potentials are required for different steps in the mechanism of the oxidation reaction. Or, pulsing to high and low potentials prevents products from the oxidation reaction from poisoning the surface of the working electrode and stopping detection. The methods used to detect these species are called Pulsed Amperometry and Integrated Amperometry.

1.2.3.1 Pulsed Amperometry

During pulsed amperometry, a repeating program of three potentials is applied to the cell. The current is measured during the first potential, after a delay to allow charging current to decay. The second and third potentials are used for the positive and negative pulses. The major application for pulsed amperometry is the detection of carbohydrates. (The Dionex Pulsed Amperometric Detector PAD-II uses this form of detection.) The PED performs Pulsed Amperometry in the **INTEGRATED AMPEROMETRY MODE**.

1.2.3.2 Integrated Amperometry

The PED employs the more advanced technique of integrated amperometry, which is used to detect species where oxidation is catalyzed by the formation of metal oxide on the working electrode's surface. The major applications are for amines and sulfur species. During integrated amperometry, a multipotential waveform is applied to the working electrode. Part of the waveform employs either a pulse or scan to a potential where oxidation of both the analyte and the working electrode surface occurs. This is followed by a return to a potential sufficient to reverse the reaction and reduce the oxide layer back to pure metal (but does not reduce the analyte). The current is integrated during the entire cycle. Since the net charge from oxide formation and reduction is near zero, the sensitivity of the baseline to mobile phase changes is limited. This method is particularly useful during gradient elution.

When the PED **MODE** is set to **INTEGRATED AMPEROMETRY**, the **ANALOG RANGE** and the detector output are in coulombs (C).

1.3 PROGRAMS FOR AMPEROMETRY AND INTEGRATED AMPEROMETRY

The parameters used in the **AMPEROMETRY** and **INTEGRATED AMPEROMETRY** modes are stored in programs. For example, the potential vs. time program applied to the working electrode must be entered into microprocessor memory. If the selected **MODE** is **INTEGRATED AMPEROMETRY**, the times during which the cell current is integrated must also be set. These parameters constitute a program.

A maximum of fifteen programs can be stored in memory. Five programs (A through E) are reserved for DC amperometry and cyclic voltammetry. Ten programs (0 - 9) are reserved for Pulsed and Integrated amperometry. The detector contains several programs, written by Dionex, which are designed to allow you to be up and running as soon as possible. These programs are described in the Programs Update (Doc. No. 034329). When you select the Dionex program that corresponds to your application, all detector parameters except **ANALOG RANGE** are automatically set.

1.4 DETECTOR CELLS

The PED is used with either the conductivity or amperometry cell.

1.4.1 CONDUCTIVITY CELL

The PED conductivity cell can be used for either suppressed or nonsuppressed (single-column) ion chromatography. The cell contains a thermistor in the flow stream to measure the temperature of the mobile phase. The temperature compensation circuitry in the detector can be used to minimize long-term baseline drift caused by temperature fluctuations. Short-term baseline noise can be reduced by using the optional Dionex Thermal Stabilizer (TS-1), P/N 041480.

1.4.2 AMPEROMETRY CELL

The amperometry cell is a three-electrode, thin-layer cell containing replaceable working and reference electrodes. The stainless-steel body serves as the counterelectrode. The following types of working electrodes are available:

- **Gold (Au).** The gold working electrode is used with integrated and pulsed amperometry to detect carbohydrates, sulfur species, amines, and amino acids.
- **Platinum (Pt).** The platinum working electrode is used to detect simple alcohols and aldehydes. Platinum can also be used to detect carbohydrates, sulfur species, amines and amino acids. Integrated and pulsed amperometry are used to detect these species. Platinum is also used with DC amperometry to detect iodide, ascorbic acid, sulfite, and other easily oxidizable species.
- **Silver (Ag).** The silver working electrode is used to detect species which form complexes or precipitates with silver. These include cyanide, sulfide, and iodide. DC amperometry is used for detection.
- **Glassy Carbon (GC).** The glassy carbon working electrode is used to detect oxidizable organic molecules such as phenols, catechols, and catecholamines. DC amperometry is used to detect these species.

Two interchangeable reference electrodes are used:

- **pH.** A combination pH-Ag/AgCl reference electrode (P/N 042899) is used for gradient elution applications in which the mobile phase pH is changed during the gradient. The use of a pH reference electrode minimizes the baseline shift caused by the changing pH. Since one-half of the pH reference electrode is a standard Ag/AgCl reference electrode, either the pH or Ag/AgCl half cell can be used on the combination electrode. The electrode can also be used to monitor the pH of the mobile phase.
- **Ag/AgCl.** (Optional) This standard silver/silver chloride reference electrode (P/N 042442) can be used for most applications.

SECTION 2
INSTALLATION

2.1 UNPACKING AND INSPECTION

Unpack the detector and inspect it for shipping damage immediately after you receive it. Because many carriers limit their claim period to 10 days after the delivery date, Dionex recommends that you report any shipping damage to the incoming carrier immediately. To request help in assessing shipping damage or to report a missing or damaged part, refer to the list below and contact the nearest Dionex office.

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2.2 FACILITIES REQUIRED

2.2.1 BENCH SPACE

Refer to the dimension specifications in Appendix A to determine the amount of bench space required for your detector. The detector can be installed in the double-height system enclosure for any of the following: the Series 2000i, Series 2000i/SP, Series 4000i, Series 4500i, or BioLC. It can also be installed in a single stand-alone enclosure. Consult your system operator's manual for further information about system bench space requirements.

2.2.2 POWER

The detector requires a grounded, single-phase power source. The three-conductor line cord provides a safety ground. The source must demonstrate a clean, sinusoidal wave form. High-voltage, high-frequency line spikes may damage the detector.

The operating voltage (100, 120, 220, or 240 volts AC at 50 or 60 Hz line frequency) must be regulated to within ±10% of the selected operating voltage. If you are unsure of the line voltage in your lab, contact local electrical personnel before installing the detector. If you need to change the selected operating voltage, refer to the instructions in Section 2.3.2.

2.3 INSTALLATION INSTRUCTIONS

2.3.1 CONTENTS OF THE SHIP KIT

Your Ship Kit (P/N 042400) contains items essential for installation. The kit includes a line cord (P/N 96708) for the 100/120 VAC power connection and wire assembly for the **RECORDER** connections. It also contains two fuses: a 250 mA sloblo fuse (P/N 954727) for 100/120 VAC operation and a 125 mA sloblo fuse (P/N 954730) for 220/240 VAC operation.

NOTE: **For 220/240 VAC operation, replace the line cord with an appropriate power cord, or strip the line cord 100/120 VAC connector and replace it with the appropriate connector. Wire color designations are for U.S. operation (Black = *Hot*, White = *Neutral*, Green = *Chassis Safety Ground*).**

Various tubing assemblies are included for the columns and cells. In addition, the Ship Kit contains a trimmer adjustment tool (P/N 035617) for calibrating the amperometry cell pH reference electrode. *(Store this tool in a convenient, secure place for future use.)* The kit also contains a 1.5-inch (3.8 cm) screw (P/N 011960) for mounting the conductivity cell.

NOTE: **If you plan to install the conductivity cell in an LCM which does not include an optional Thermal Stabilizer, you need a #6 hex nut to mount the cell on the center wall support. This nut is provided in the LCM Ship Kit.**

2.3.2 OPERATING VOLTAGE SELECTION

Before installing the AC line cord, locate the fuse compartment on the rear panel of the detector (Figure 2-1). Refer to Figure 2-2 and then follow the numbered steps listed below that figure to set the detector's operating voltage to match the line voltage you are using.

Figure 2 - 1
Detector Rear Panel

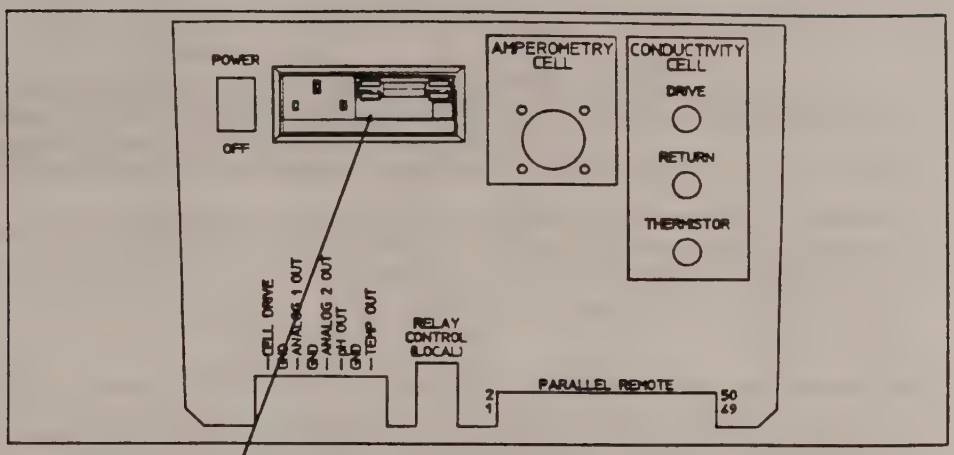
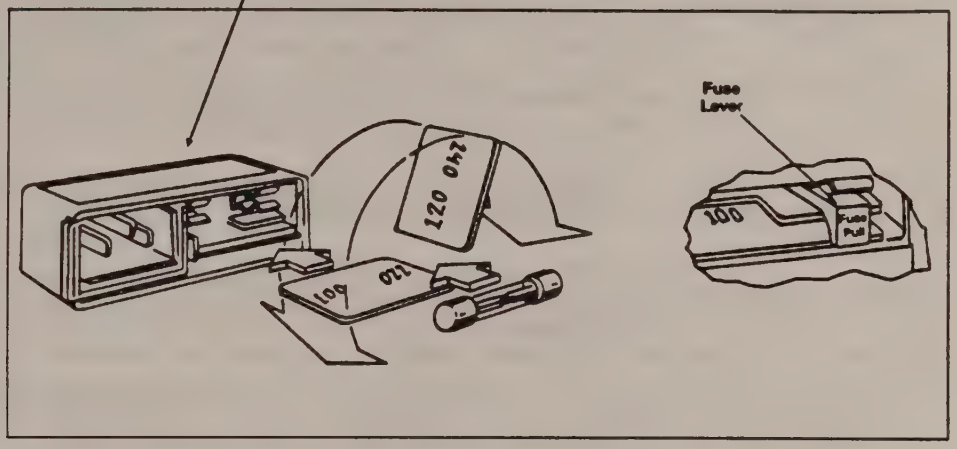


Figure 2 - 2
Changing the Selected Operating Voltage



1. Make sure the **POWER** switch is set to the **OFF** position.
2. Slide the clear window covering the fuse compartment to the left.
3. Rotate the Fuse Pull lever to the left and remove the fuse.
4. Pull the voltage select card out of the fuse compartment, using a pair of needle nose pliers.
5. Orient the card so that the voltage you intend to use is displayed on the upper left of the card.
6. Reinsert the card so that the desired voltage marker is visible. Push the Fuse Pull lever to the right again.
7. Replace the fuse with the appropriate part:

   250 mA (P/N 954727) for 100/120 VAC operation
   125 mA (P/N 954730) for 220/240 VAC operation

NOTE: The detector is shipped with a 250 mA sloblo fuse. If your line voltage is 220/240 AC, change the fuse to a comparable 125 mA fuse before connecting line power.

8. Slide the compartment window to the right, exposing the line cord connector.

9. Firmly seat the line cord on the connector.

NOTE: **For 220/240 VAC operation, replace the line cord with an appropriate power cord, or remove the line cord 100/120 VAC connector and replace it with the appropriate connector, keeping in mind that wire color designations are for U.S. operation (Black = *Hot*, White = *Neutral*, Green = *Chassis Safety Ground*).**

10. Plug the cord into an AC power outlet or any appropriate power source that supplies a true ground.

11. Turn **ON** the **POWER** switch.

2.3.3 CONDUCTIVITY CELL INSTALLATION

The Chromatography Module sub-manual in the System Operator's Manual gives instructions for installing the conductivity cell. Connect the cell into the flow path after the suppressor. The flow path through the cell must be from the bottom (inlet) to the top (outlet) to prevent bubble trapping. When installing the cell, make sure the cell is oriented so that the thermistor cable, which is in the center and is slightly offset from the cell cables, is up. This ensures that the liquid stream passes the thermistor after it passes the electrodes. After installing the cell in the Chromatography Module, connect the cell cables to the detector.

1. Thread the detector cables through the slot or opening in the rear wall of the Chromatography Module, one connector at a time.

NOTE: **If you use an Advanced Chromatography Module, thread the cell cables through the top slot and then down behind the electronics chassis and into the lower rear compartment.**

2. Route the cell cables from the rear of the Chromatography Module to the rear of the detector.

3. One of the cables has a white band marked "T" near the connector. Connect this cable to the **CELL THERMISTOR** connector on the rear panel (Figure 2-1).

4. Connect one of the remaining cables to the **CELL DRIVE** connector. Connect the other cable to the **CELL RETURN** connector.

2.3.4 AMPEROMETRY CELL INSTALLATION

2.3.4.1 Polishing the Working Electrode

The working electrode must be polished before it is installed in the amperometry cell. To maintain sensitivity after the initial installation, polish the working electrode surface whenever you observe high background current, a decrease in sensitivity, or other degradation in the detector output.

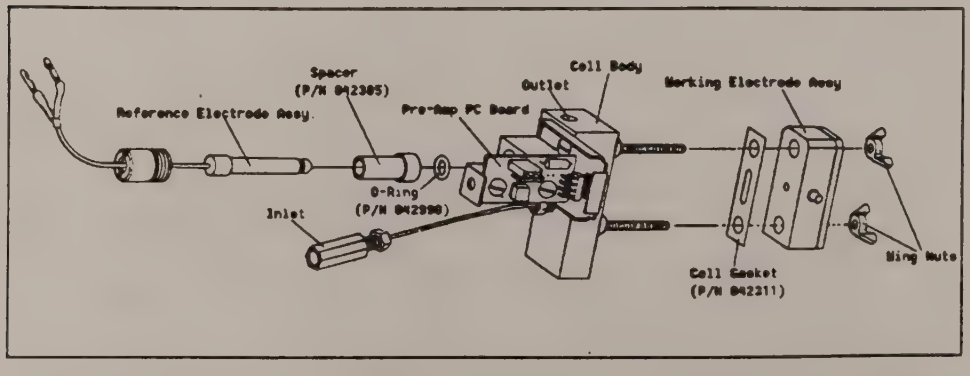


Figure 2-3
Amperometry Cell Assembly

1. Unscrew the wing nuts holding the working electrode to the cell body (Figure 2-3), and carefully separate the parts.

CAUTION: Handle the cell gasket and the inside surfaces of the cell carefully, to prevent scratches which may cause leaks.

2. Locate the polishing kit (P/N 036313) in the Ship Kit. The kit contains two polishing pads, a bottle of fine polishing compound, and a bottle of coarse polishing compound. Using indelible ink, mark the plastic side of each pad to designate whether it is to be used with the coarse or fine polishing compound. Also mark the fine pad to designate which working electrode type it is to be used with.

NOTE: **Polishing more than one type of working electrode with the same fine polishing pad may contaminate the electrode surface with microparticles from the other working electrodes. A separate polishing pad is shipped with each type of working electrode. Using indelible ink, mark each pad to indicate the working electrode with which it is used.**

3. Moisten the plastic side of the fine polishing pad slightly with water and place it on a smooth, flat surface.

4. Sprinkle about one-half gram of fine polishing compound (P/N 036316) in the center of the suede side of the polishing pad. Add enough deionized water to make a thick paste.

5. Using the working electrode block, spread the paste evenly over the pad. Then, using firm pressure and a circular motion, polish the surface of the electrode block for approximately one minute. Add more water sparingly if the pad dries out while polishing.

6. Use deionized water to rinse off all polishing compound from the electrode block. An ultrasonic cleaner is effective for thoroughly cleaning the electrode block. Carefully wipe the surface of the block with a soft damp cloth or damp paper towel. Inspect the surface of the working electrode to make sure it is clean.

NOTE: **The pads are reusable. Do not rinse the polishing compound from the pads after polishing is complete. After the initial use, add only enough polishing compound to maintain the coating on the pad.**

NOTE: **During the normal operating life of the working electrode, the surface will gradually become pitted. If you notice any degradation in performance, such as a noisy baseline or tailing peaks, polish the working electrode, using the coarse polishing compound. Then, rinse the electrode thoroughly, wipe the coarse polishing compound from the surface of the electrode, and polish with the fine polishing compound. You may use the pad designated for use with coarse polishing compound on all electrodes.**

2.3.4.2 Assembling and Installing the Amperometry Cell

The amperometry cell is installed directly after the column. No suppressor is used. A second detector such as a UV/Visible Detector may be installed in-line after the amperometry cell as long as the pressure at the amperometry cell inlet is less than 100 psi. Because of the volume within the reference electrode section of the cell, there may be some band broadening noticed at the second detector.

1. Inspect the cell gasket for scratches or damage. If scratched or damaged, replace the gasket (P/N 042311). Rinse the gasket with deionized water, and clean the polished surface of the cell with a damp paper towel. Install the gasket over the alignment studs on the cell block.

2. Install the working electrode block over the alignment studs and fasten it in place with the wing nuts.

NOTE: Working electrode blocks are symmetrical and can be installed in either direction.

3. Feed the reference electrode cable through the reference electrode cell cap (P/N 042680).

4. Connect the reference electrode cable to the cell preamp PC board. Detach the Cell Cover (P/N 042303) from the cell to gain access to the cell preamp board. If you are using an Ag/AgCl reference electrode, connect it to the pin labeled **Ag**. If you are using a pH reference electrode, connect the reference electrode cable center conductor (the one with red insulation) to the pin labeled **pH** on the preamp board. Connect the shield with black insulation to the pin labeled **Ag**. Do not insert the reference electrode into the cell yet.

5. Make sure the detector **CELL** is turned **OFF**. Connect the amperometry cell cable to the PED rear panel. Extend the cable assembly from the rear of the detector to the cell, and plug the cable into the multi-pin connector on the cell. (One pin has been removed and its position in the connector blocked so that the connector can only be attached in one way.)

6. Remove the protective boot from the reference electrode and rinse the electrode in deionized water. Be sure to rinse off any precipitated salt.

7. If you are using a pH reference electrode, calibrate the reference electrode (see Section 2.3.4.3).

8. Place the spacer (P/N 042305) and then an o-ring (P/N 042990) on the reference electrode. Insert the reference electrode, with the o-ring and spacer, into the cell and hand tighten the cap. **DO NOT OVERTIGHTEN. DO NOT USE PLIERS.**

9. Carefully coil the reference electrode cable and attach the cell cover to the cell. Hand tighten the screw.

10. Using the working electrode wire with **#1** marked on the insulation, connect the amperometry cell cable to the working electrode.

11. Attach the tubing from the column to the cell inlet (see Figure 2-3).

12. The short length of stainless-steel tubing at the cell inlet is an integral part of the cell. DO NOT REMOVE IT. This tube extends the counterelectrode, thereby shielding the working electrode from electrical noise.

    When using a stainless-steel-based HPLC, connect the stainless-steel outlet tube from the column to the plastic coupler (P/N 042627), and not directly to the cell inlet. This is necessary to electrically isolate the pump (which may be grounded) from the counterelectrode (which is the cell body).

    When using Thermoflare 1/4-28 fittings, first cut off the existing fitting on the column outlet tubing, then slide a 10-32 bolt followed by a ferrule onto the tubing. Screw this into the coupler. Install the ferrule and fitting in accordance with Dionex Pub. No. 034213.

13. Connect the waste line to the cell outlet.

14. Mount the amperometry cell in your chromatography module, using the bracket you ordered at the time you ordered your detector. Install in accordance with the instructions included with the bracket.

Chromatography Module	Mounting Bracket Kit Part Number
Liquid Chromatography Module (LCM)	P/N 042188
Advanced High-Pressure Chromatography Module (CHA)	P/N 042186
Basic High-Pressure Chromatography Module (CHB)	P/N 042187
Bench (freestanding cell)	P/N 042185

NOTE: The purpose of the mounting bracket is to orient the cell with the outlet pointing up, thus ensuring that bubbles are swept out. Although it is possible to operate the detector with with the cell simply lying on its side on a flat surface, that configuration makes it easier for bubbles to become trapped in the cell.

2.3.4.3 Calibrating the pH Reference Electrode

NOTE: If you are using the Ag/AgCl reference electrode, omit this procedure.

pH electrodes must be calibrated in order to produce accurate pH readings. This is accomplished on the PED by adjusting the slope and the offset. Although it is possible to calibrate the electrodes by pumping pH buffers through the cell, Dionex recommends that you follow the procedure below, in which calibration is performed with **CELL OFF** and the pH reference electrode removed from the cell.

NOTE: Calibrating the pH electrode in the cell with the CELL powered ON may cause oxidation or reduction of some components of the pH calibration buffers, possibly causing poisoning of the working electrode surface. If this occurs, restore the working electrode by polishing (see Section 2.3.4.1).

1. Verify that the amperometry cell cable is connected to the cell and to the detector. Verify that the connectors on the pH reference electrode are properly connected to the cell preamp PC board (see Section 2.3.4.2).

2. Make sure that **CELL** is **OFF**. Set the **DISPLAY** to **pH/TEMP**.

3. Remove the protective boot from the pH electrode and rinse the electrode in deionized water. Be sure to rinse off any precipitated salt.

4. Place the electrode in a buffer solution of pH 7.

5. Using the trimmer adjustment tool or a small screwdriver, adjust the pH OFFSET trimpot on top of the detector until the display reads 7.0.

6. Remove the pH reference electrode from the pH 7 buffer, rinse it with deionized water, and transfer it to a solution with known pH near the expected pH of your mobile phase. For acidic mobile phases, pH 4 buffer is usually sufficient, while pH 10 buffer can be used for calibration when basic mobile phases are used.

7. Adjust the pH SLOPE trimpot until the display indicates the pH of the buffer.

8. Rinse the electrode and insert it into the cell, using the procedure listed in Section 2.3.4.2, Step 8.

NOTE: **Readings from the pH reference electrodes are accurate at most mobile phase pH values. However, at very high pH (generally above pH 12), it is normal for the measured pH to begin reading lower than the actual pH. This is called "alkaline error".**

2.3.5 RECORDER CONNECTION

2.3.5.1 Analog Output

Using the wire assembly from the Ship Kit, connect the recorder or integrator to the detector rear panel (Figure 2-1) as follows: Connect the red wire to **ANALOG 1 OUT** and the black wire to a ground (**GND**).

2.3.5.2 pH and Temperature Outputs

If desired, you can connect a second input on a dual-pen recorder to the pH or temperature analog outputs on the detector rear panel. Alternatively, separate analog recording devices can be used. Connect the red wire from the wire assembly to **pH OUT** or **TEMP OUT**, and the black wire to a ground (**GND**).

2.3.6 RELAY REMOTE CONNECTION

Relay Control uses simple TTL logic levels or relay contact closures to control three detector functions: **AUTO OFFSET** (Function 1), **ANALOG RANGE UP** (Function 2), and **ANALOG RANGE DOWN** (Function 3). These functions allow you to use a remote controller or timed event function of an integrator to offset the background and increase or decrease the output range. You must use the front panel switches to control all the remaining detector functions.

Connect the telephone-style connector end of the relay cable to the **RELAY CONTROL** connector on the detector rear panel (Figure 2-1). For each Relay Control function to be controlled, connect the corresponding connector on the other end of the cable to a contact-closure relay on the Timed Event Module of an integrator or other system equipped with TTL or contact-closure relay control outputs.

See Appendix B for a description of the Relay Control connections.

2.3.7 PARALLEL REMOTE CONNECTION

To use Parallel Remote, connect a 50-pin parallel interface cable between the **PARALLEL INTERFACE** connector on the detector rear panel (Figure 2-1) and a Dionex Computer Interface, or any suitably-equipped parallel interface system. Refer to the interface manual for complete installation instructions. Ensure that the ribbon cable edge that has the colored wire is near the pin 1 marking at the left of the **PARALLEL REMOTE** connector label on the PED rear panel. See Appendix C for a description of the Parallel Interface connections.

SECTION 3
OPERATION

This section presents the procedures and other information required to operate the PED. When using this section you may find it useful to refer to Section 4 for detailed illustrations and explanations of PED controls, displays and connectors.

3.1 CONDUCTIVITY

When the PED **MODE** is set to **CONDUCTIVITY**, the detector is ready to use. Programs are not used in the **CONDUCTIVITY MODE**, so the **PROGRAM** display on the front panel is blank. With the exception of setting the **ANALOG RANGE**, the only detector parameters affecting the **CONDUCTIVITY MODE** are the temperature compensation factor and the cell calibration. Both of these are preset at the factory before shipment. Since there are minor differences in temperature compensation factors between mobile phases, you should check to see if the factory preset value of 1.9%/°C is optimum for the mobile phase you are using. Use the procedure listed below in Section 3.1.1 to optimize the temperature compensation factor. The preset cell calibration probably does not need to be adjusted. To check that it is still correct, follow the procedure in Section 3.1.2.

3.1.1 OPTIMIZING TEMPERATURE COMPENSATION

Temperature compensation corrects the changes in response caused by temperature changes in the mobile phase. This greatly minimizes baseline drift caused by laboratory temperature changes and also improves peak reproducibility. The magnitude of the compensation is set by the temperature compensation factor, which is the change in conductivity as a function of temperature, in percent per degree C.

The conductivity of most aqueous solutions rises approximately 2% per degree C. Use the following procedure to measure the change in conductivity as a function of temperature and to set the temperature compensation factor.

1. Begin with the chromatograph set up and ready for use. Turn the pump, the regenerant (if using a suppressor), and the detector cell **ON**.

2. Set **DISPLAY** to **pH/TEMP**. Scroll the select buttons down to set the temperature compensation factor to 0.0. Press **ENTER**.

3. Set **DISPLAY** to **TOTAL**. Set the **ANALOG RANGE** to the range closest to 10% of the total conductivity. Press **AUTO OFFSET**. Allow the system to stabilize and the baseline to steady.

4. Record the total conductivity.

5. Set **DISPLAY** to **pH/TEMP**. Record the cell temperature.

6. Warm the inlet line to the conductivity cell by holding it in your hand. Watch the cell temperature rise. Wait for the cell temperature and recorder output (if you are using a recorder) to stabilize. Record the new cell temperature.

7. While still holding onto the cell inlet line, set **DISPLAY** to **TOTAL**. Record the new total conductivity. Let go of the inlet line.

8. Calculate the percent change in total conductivity.

$$\% \text{ change} = \frac{\text{final conductivity - initial conductivity}}{\text{initial conductivity}} \times 100$$

9. Calculate the temperature compensation factor by dividing the percent change in total conductivity by the change in temperature.

10. Set **DISPLAY** to **pH/TEMP**. Scroll the select buttons up to set the temperature compensation factor to the calculated value. Press **ENTER**. Press **AUTO OFFSET**.

11. Warm the inlet line to the cell and note the total change in conductivity after the cell has equilibrated at a new temperature.

NOTE: **The temperature compensation circuitry corrects the measured conductivity at a rate of once per second. Rapid changes in temperature, such as those caused by this procedure, produce sharp baseline changes. This is not a problem during normal operation because temperature does not change this fast during normal analysis.**

12. If the final (warm) conductivity is higher than the initial (cool) conductivity, raise the temperature compensation factor by 0.1 and repeat Step 11. Conversely, if the conductivity drops during Step 11, lower the temperature compensation factor. Repeat step 11 until the factor producing the least change in conductivity is found.

3.1.2 CALIBRATING THE CONDUCTIVITY CELL

The conductivity cell is calibrated by pumping a solution of known conductivity through the cell and adjusting the detector output to the known value. A 1 mM solution of potassium chloride is used in the procedure described below. The following method uses a syringe to pump the calibration solution through the cell. Since conductivity is somewhat affected by the flow rate, you may wish to use the pump on your chromatography system to calibrate the cell at the flow rate you normally use for your application. The advantage of using a syringe is that it avoids the possibility of contaminating the chromatography flow system with KCl. Actually, minor errors in cell calibration are of little importance since quantitative analysis is generally performed using standard solutions, and the errors are cancelled out.

Use the following procedure to calibrate the cell.

1. Prepare a 10 mM KCl solution by dissolving 0.7455 g of KCl in 1 L of 18 MΩ deionized water in a volumetric flask. (For greatest accuracy, the KCl should be desiccated.)

2. Prepare a 1 mM KCl solution by diluting the 10 mM solution 1 in 10.

3. Fill a clean syringe with the 1 mM KCl solution. Using a short piece of tubing, connect the syringe to the cell inlet.

4. Set the **MODE** to **CONDUCTIVITY**. Set **DISPLAY** to **pH/TEMP**, and ensure that the temperature compensation factor is set to 1.9 (Section 3.1.1).

5. On S1 (see Figure 4-3), set position 1, **COND CELL CALIB**, to the **ON** position. The letter c will appear in the **PROGRAM** display.

6. Turn the cell **ON**. The **DISPLAY** will be in the **TOTAL** position. While forcing solution through the cell with the syringe, scroll the **SELECT** buttons under the display up or down until the display reads **147.0 µS**. Press **ENTER** to set the cell calibration.

7. Reset the detector for normal operation by turning position 1 on S1 **OFF**.

3.2 DC AMPEROMETRY

To operate the PED in the DC Amperometry mode, it must first be programmed. Five programs (A through E) are reserved for the **AMPEROMETRY** mode and can be used either for DC amperometry or cyclic voltammetry. Enter, edit, or delete programs using the procedures described in this Section.

DC amperometry programs are very simple, containing only a single time and a single potential.

3.2.1 ENTERING A NEW PROGRAM

1. Set the **MODE** to **AMPEROMETRY** and the **DISPLAY** to **TIME/POTEN**.

NOTE: You cannot change the mode unless CELL is in the OFF position.

2. Select an empty **PROGRAM**. ("Empty" programs actually contain a single program step with the time at 0.00 seconds and the potential at 0.00 volts. Pressing **LIST** does not show steps at a later time.)

3. Press **ENTER** to accept 0.00 s as the program step to be edited.

4. 00.0 s and 0.00 V are displayed and V is blinking. Use the **SELECT** buttons under the display to scroll to the desired applied potential.

5. Press **ENTER** to accept the new potential.

6. Turn the cell **ON**, or if the cell is already **ON**, turn it **OFF** and then immediately **ON** again. Turning on the cell stores the program in memory. The program can be recalled at any time by selecting the program letter.

3.2.2 EDITING A PROGRAM

The applied potential can be changed at any time by using the following steps.

1. Select **TIME/POTEN**.

2 Press **ENTER**.

3. Scroll the **SELECT** buttons to the desired potential.

4. Press **ENTER**.

5. Turn the cell **OFF**, and then **ON**.

3.2.3 DELETING A PROGRAM

To delete an entire program, display the Time Zero step and press the Delete key. The message DELETE PGM? is displayed. Press the Enter key to delete the program. Pressing any other key deletes the message without deleting the program.

NOTE: After programming the PED, follow the instructions in Section 2.3.4, *Amperometry Cell Installation*, and either Section 3.6, *Manual Operation*, or 3.7 *Remote Operation*.

3.3 CYCLIC VOLTAMMETRY

Cyclic voltammetry is performed in the **AMPEROMETRY** mode, using the amperometry cell. With the pump **ON** and the mobile phase flowing through the cell, this technique produces results similar to those obtained by rotated disk voltammetry in a standard beaker cell. With the flow off, rapid depletion of analyte next to the working electrode is typical of thin layer voltammetry.

3.3.1 RECORDER CONNECTIONS

To perform cyclic voltammetry, an X-Y recording device is used. Standard X-Y recorders with sweep rates of up to about 0.25 V/s can be used. At faster sweep rates, either a storage oscilloscope or a computer system must be used. Connect the **CELL DRIVE** output on the rear of the detector to the X input on the recording device, and the **ANALOG 1 OUT** to the Y input on the recording device. You can use a chart recorder or other device which plots current vs. time. However, using such a device makes interpreting the voltammogram more difficult.

NOTE: **The Y Analog output from the PED is not bipolar. To record both negative and positive currents, you must set the baseline level to 50% of full scale. Do this by turning ON positions 6 and 8 on S2 (accessible from the top cover). When zeroing your recording device, remember that zero current will now be recorded at 50% of full scale.**

3.3.2 NORMAL AND RAPID SCAN CYCLIC VOLTAMMETRY

Cyclic voltammetry can be performed in either the Normal Scan or Rapid Scan modes. Position 2 on S1 determines the mode. In Normal Scan (position 2 **OFF**), programs up to 99.9 s can be used. Data is collected at a rate of 20 measurements per second, or one measurement every 50 ms. In Rapid Scan (position 2 **ON**), data is collected at a rate of 100 measurements per second, or one measurement every 10 ms. Total program length is limited to 9.99 s.

The PED is capable of performing rapid scan cyclic voltammetry, with sweep rates of several volts per second. Rapid scan cyclic voltammetry is performed on the same time scale as integrated and pulsed amperometry, and therefore is very useful for developing methods using these techniques. To perform rapid scan cyclic voltammetry, turn position 2 of S1 **ON**.

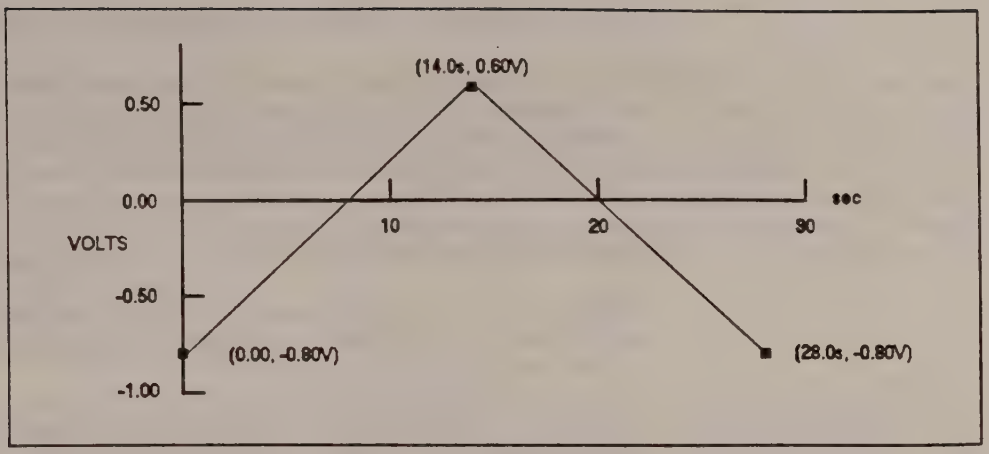
NOTE: **When performing rapid scan cyclic voltammetry, peak currents may occur which are in excess of the maximum current the detector is capable of measuring. This is a common occurrence when the applied potential reaches the potential limits. Although the current may still be supplied to the cell (maximum is 10 mA), currents in excess of 3 mA cannot be measured. Current greater than 3 mA causes the warning message "CELL OVERCURRENT" to appear. To delete the message, press DELETE. If the message continues to appear, turn OFF position 3 on S1. With position 3 OFF, the message appears only during the time that the cell current is in excess of 3 mA, and is automatically deleted when the current drops below 3 mA.**

3.3.3 ENTERING A NEW PROGRAM

To perform cyclic voltammetry on the PED, it must first be programmed. Five programs (A through E) are reserved for the **AMPEROMETRY** mode and can be used either for DC amperometry or cyclic voltammetry. Enter new programs using the procedures described in this Section. Programs may also be edited or deleted, as described in Sections 3.3.4 and 3.3.5.

Unlike DC amperometry, a time/potential program must be entered for cyclic voltammetry programs. The program is defined by points on an X-Y graph, where time is the X axis and potential is the Y axis. See Figure 3-1 for an example.

Figure 3 -1
Example of a Program for Cyclic Voltammetry



In this example the potential is cycled between -0.80 and +0.60 at a sweep rate of 0.1 V/s. Cyclic voltammetry programs contain three steps, with the potential at the first and third steps the same. The first program step at 0.00 s is the initial potential of -0.80 V. To calculate the time for the second program step, divide the total voltage scanned by the desired sweep rate.

$$\frac{\text{Total Voltage Scanned}}{\text{Sweep Rate}} = \frac{1.4\ \text{V}}{0.1\ \text{V/s}} = 14\ \text{s}$$

The time for the second program step is 14 s. The third program step is an equal interval later, or 28 s.

Program steps are entered by first entering the time, followed by the potential. The first step of every program is at time zero. Since this cannot be changed, the first value to be entered is the first potential. The remaining times and potentials are entered in sequence until the program has been completed. Use the following steps to enter a new program for cyclic voltammetry.

1. Set position 2 on S1 (see Section 4.3) to either **OFF** (Normal Scan) or **ON** (Rapid Scan). The choice depends on the length of your program and the resolution desired. In Normal Scan, the maximum program length is 99.9 s, and the resolution is 0.05 s (20 Hz sample rate). In Rapid Scan, the maximum program length is 9.99 s and the resolution is 0.01 s (100 Hz sample rate).

2. Set the **MODE** to **AMPEROMETRY** and the **DISPLAY** to **TIME/POTEN**.

NOTE: You cannot change the mode unless CELL is in the OFF position.

3. Select an empty **PROGRAM**. ("Empty" programs actually contain a single program step with the time at 00.0 seconds and the potential at 0.00 volts. Pressing **LIST** does not show steps at a later time.)

4. Press **ENTER** to accept 00.0 s as the program step to be edited.

5. 00.0 s and 0.00 V are displayed, and V is blinking. Scroll the **SELECT** buttons under the display to set the first potential. When the potential is set to the desired value, press **ENTER**.

6. The time of 00.0 s is now blinking, indicating that you should set the second time. Using the **SELECT** buttons, scroll to the time of the second program step and press **ENTER**.

7. Scroll to the potential of the second program step and press **ENTER**.

NOTE: **The available potentials are limited to ensure that the change in potential during each measurement period (0.05 sec in Normal Scan, 0.01 in Rapid Scan) is an integer number of millivolts. If the desired potential is not available, change the time interval so that the resulting sweep rate will be a multiple of either 0.02 V/s (Normal Scan) , or 0.10 V/s (Rapid Scan).**

8. Use the same method as detailed above to set the third time and potential.

9. Turn the cell **ON**, or if the cell is already **ON**, turn it **OFF** and then immediately **ON** again. Turning on the cell stores the program in memory. It can be recalled at any time by selecting the program letter.

NOTE: **To list the steps in a program, set the DISPLAY to TIME/POTEN and press LIST. The steps from the example program shown above would be displayed in sequence as follows:**

00.0 s - 0.80 V
14.0 s +0.60 V
28.0 s - 0.80 V

3.3.4 EDITING PROGRAMS

To delete a program step, press DELETE.

NOTE: **The first program step at time = 00.0 s cannot be deleted.**

To change a time:

1. **LIST** to the program step to be edited and press **DELETE**.

2. Scroll to the new time and press **ENTER**.

3. Scroll to the desired potential and press **ENTER**.

NOTE: **If you edited any program step other than the last, LIST through the program and ensure that all intervals after the edited step use acceptable sweep rates. (See NOTE after step 7, above.)**

4. Turn the cell **OFF**, then **ON** again.

To change a potential:

1. **LIST** to the program step to be edited.

2. Press **ENTER** to keep the displayed time.

3. Scroll to the desired potential and press **ENTER**.

4. Turn the cell **OFF**, then **ON** again.

3.3.5 DELETING A PROGRAM

To delete an entire program, display the Time Zero step and press the Delete key. The message DELETE PGM? is displayed. Press the Enter key to delete the program. Pressing any other key deletes the message without deleting the program.

3.3.6 AUTO OFFSET

After programming the detector, turning on the cell stores the program in memory and begins applying the program to the cell. In cyclic voltammetry a baseline offset is generally not used, so no offset is applied to the output when the cell is turned on. However, pressing **AUTO OFFSET** subtracts the instantaneous value of the current from all subsequent measurements, offsetting the entire voltammogram. This can be useful when a more sensitive range is used to amplify either the positive or negative portion of the voltammogram. Of course, the other half of the voltammogram is off scale and is not recorded. To cancel **AUTO OFFSET**, turn the cell **OFF** and then back **ON** again.

3.4 INTEGRATED AMPEROMETRY

The **INTEGRATED AMPEROMETRY** mode is used for both integrated amperometry and pulsed amperometry. To perform these methods a program must be selected. Programs 0 through 9 are reserved for integrated and pulsed amperometry. You can use existing programs, or you can enter, edit, or delete programs.

3.4.1 USING EXISTING PROGRAMS

The detector already contains several programs written by Dionex that should enable you to be up and running quickly. These programs are described in the Programs Update (Doc. No. 034329). When you select the Dionex program corresponding to your application, all detector parameters except **ANALOG RANGE** will be automatically set. To write a new program or to change any of the parameters in a program, use the procedures in the following paragraphs.

3.4.2 ENTERING A NEW PROGRAM

To create your own program for integrated or pulsed amperometry, you need to enter both a time/potential program and an integration period. The repeating sequence of potentials applied to the cell working electrode is defined by points on an X-Y graph, where time is the X axis and potential is the Y axis. See Figure 3-2 for an example.

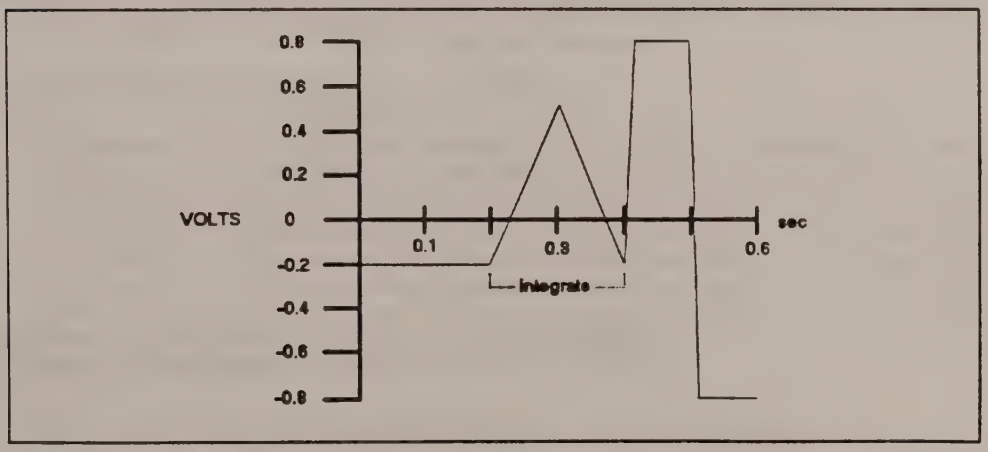


Figure 3-2 Example of a Program for Integrated Amperometry

When entering program steps, enter the time first and then the potential. The first step of every program is at time zero. Since this cannot be changed, the first value to be entered is the first potential. The remaining times and potentials are entered in sequence until the program has been completed. To enter a new program:

1. Set the **MODE** to **INTEGRATED AMPEROMETRY** and the **DISPLAY** to **TIME/POTEN**.

NOTE: You cannot change the mode unless CELL is in the OFF position.

2. Select an empty **PROGRAM**. ("Empty" programs actually contain a single program step with the time at 0.00 seconds and the potential at 0.00 volts. Pressing **LIST** does not show steps at a later time.)

3. Press **ENTER** to accept 0.00 s as the program step to be edited.

4. 0.00 s and 0.00 V are displayed, and V is blinking. Scroll the **SELECT** buttons under the display to set the first potential. When it is set to the desired value, press **ENTER**.

5. The time of 0.00 s is now displayed and the s is blinking, indicating that the next parameter to set is the second time. Using the **SELECT** buttons, scroll to the time of the second program step and press **ENTER**.

6. Scroll to the potential of the second program step and press **ENTER**.

7. Use the same method to set the third and all remaining times and potentials.

8. Set the **DISPLAY** to **INTEGRATE**.

9. Set the beginning of the integration period and press **ENTER**.

10. Set the end of the integration period and press **ENTER**.

NOTE: **Most programs will contain only one integration period. To enter multiple integration periods, enter the beginning and ending times in sequence. The charge from each of the integration periods will be added to the total.**

11. Turn the cell **ON**, or if the cell is already **ON**, turn it **OFF** and then immediately **ON** again. Turning on the cell stores the program in memory. It can be recalled at any time by selecting the program number.

12. To list the time/potential program steps, set the **DISPLAY** to **TIME/POTEN** and press **LIST**. The steps from the example program shown in Figure 3-2 would be displayed in sequence as follows:

0.00 s	- 0.20 V
0.20 s	- 0.20 V
0.30 s	+0.50 V
0.40 s	- 0.20 V
0.41 s	+0.80 V
0.50 s	+0.80 V
0.51 s	- 0.80 V
0.60 s	- 0.80 V

The integration times can be listed by setting the display to **INTEGRATE** and pressing **LIST**. The times from the example program shown in Figure 3-2 would be displayed as:

0.20 s 0.40 s

3.4.3 EDITING A PROGRAM

To delete a program step, press **DELETE.**

NOTE: **The first program step at time = 0.00 s cannot be deleted.**

To change a time:

1. **LIST** to the program step to be edited and press **DELETE.**

2. Scroll to the new time and press **ENTER**.

3. Scroll to the desired potential and press **ENTER**.

4. Turn the cell **OFF**, then **ON**.

To change a potential:

1. **LIST** to the program step to be edited.

2. Press **ENTER** to keep the displayed time.

3. Scroll to the desired potential and press **ENTER**.

4. Turn the cell **OFF**, then **ON** again.

To change an integration period:

1. Set the **DISPLAY** to **INTEGRATE**.

2. **LIST** to the integration period to be edited and press delete.

3. Enter the new integration period.

4. Turn the cell **OFF**, and then **ON**.

NOTE: Be sure to press LIST to ensure that only one (or the correct number) integration period is entered.

3.4.4 DELETING A PROGRAM

To delete an entire program, display the Time Zero step and press the Delete key. The message DELETE PGM? is displayed. Press the Enter key to delete the program. Pressing any other key deletes the display without deleting the program.

3.5 COMBINATION pH-Ag/AgCl REFERENCE ELECTRODE

The combination pH-Ag/AgCl reference electrode (P/N 042899) contains two reference electrodes in one package. The Ag/AgCl half-cell is used for most applications. The purpose of the pH half-cell is to minimize changes in the baseline during a pH gradient. Reference electrode selection is determined by the settings on S4 (accessible from the top cover). Positions 1 and 2 select the pH reference, and position 3 selects the Ag/AgCl reference. (See Section 4.3.4 for a discussion of the settings.) The pH reference electrode also reports the mobile phase pH, regardless of which half-cell is being used for the reference potential. If you do not need to use the pH half of the reference electrode, you can instead use a reference electrode which contains only an Ag/AgCl half-cell (P/N 042442).

3.5.1 ADJUSTING POTENTIALS WITH THE pH REFERENCE ELECTRODE

The potentials at which many redox reactions take place are pH dependent, with the potential shifting -0.059 V per pH unit. This is true for metal oxide formation and reduction reactions. Since the reference potential of the pH electrode also shifts by -0.059 V per pH unit, pH dependent potential shifts at the working electrode are cancelled. At a mobile phase pH of 7, the reference potential of the pH electrode is the same as that of the Ag/AgCl electrode. As the mobile phase pH is increased, the reference potential decreases approximately 0.059 V per pH unit. For example, at a mobile phase pH of 12, the reference potential of the pH electrode is -0.295 V relative to the Ag/AgCl reference electrode. At pH 12, the potentials applied to the working electrode must

be raised by approximately this amount when switching from the Ag/AgCl reference to the pH reference. In acidic mobile phases, the reference potential of the pH electrode is positive with respect to the Ag/AgCl reference, and all applied potentials must be decreased by 0.059 V per pH unit when switching from the Ag/AgCl reference to the pH reference. In basic mobile phases, the pH electrode reference potential is negative with respect to the Ag/AgCl reference, and all applied potentials must be increased by approximately 0.059 V per pH unit.

3.5.2 ALKALINE ERROR

At high pH values, the response of glass pH electrodes changes by less than -0.059 V per pH unit. This is called *alkaline error*. The 16-position rotary switch S3 (see Section 4.3) allows corrections to be made to the measured voltage response. Increasing the gain from the default value of 1.0 (position 5) provides a linear correction for alkaline error. Note that the applied potentials to the cell must be corrected. To calculate the new potentials, multiply the correction based on -0.059 V per pH unit by the gain. For example, if the gain is set to 1.5 (position A) and the displayed pH is 12.0, then the applied potentials should be increased by 0.45 V over the potentials used with an Ag/AgCl reference electrode. The switch positions and the corresponding gains are listed on the label on the top cover.

NOTE: **Corrections made with the 16-position rotary switch S3 are only active when S4 is set to position 2, pH and Ref. (See Section 4.3).**

3.6 MANUAL OPERATION

The PED must be programmed to perform either amperometry or integrated amperometry. (See sections 3.2 and 3.4 for instructions for these modes.) No programming is necessary for the PED to operate in the conductivity mode; once the front panel **MODE** switch is set to **CONDUCTIVITY**, the detector is ready for use. The only parameter which can be set is the temperature compensation factor.

The following procedure describes the basic steps for manual (i.e., front panel) operation of a detector in **LOCAL** mode. When powering up the detector, and, in most cases, when returning from **REMOTE** mode, the mode is **LOCAL**, and **CELL** is **OFF**.

1. Set **MODE**

2. Select **PROGRAM** if **AMPEROMETRY** or **INTEGRATED AMPEROMETRY** is selected.

3. Edit program, if necessary.

4. Select an **ANALOG RANGE** which will allow you to monitor the baseline on the recorder.

5. Select **CELL ON**. Once the chromatographic system is equilibrated and the baseline is stable, select the **ANALOG RANGE** sensitivity required for your analysis.

6. Press **AUTO OFFSET** just before injecting the sample.

NOTE: **The baseline may be reset to the offset level at any time by momentarily pressing AUTO OFFSET.**

3.7 REMOTE OPERATION

3.7.1 RELAY CONTROL OPERATION

This section lists the basic steps for operating the PED via Relay Control. You can use any number of the Relay Control functions. See Appendix B for a description of the Relay Control connections. The Relay Control Functions are as follows: FUNCTION 1 = AUTO OFFSET; FUNCTION 2 = RANGE UP; FUNCTION 3 = RANGE DOWN.

Use the following procedure to operate the PED using Relay Control.

1. Set **LOCAL/REMOTE** to **LOCAL**.

2. Set **MODE**, **PROGRAM**, and **ANALOG RANGE**, as described in Section 3.6, Manual Operation.

3. Turn **CELL ON**.

4. Activate **AUTO OFFSET** (Function 1) by turning **ON** and then **OFF** the corresponding relay. The **AUTO OFFSET** indicators on the front panel will change to reflect the current status.

5. To increase the **ANALOG RANGE** setting (Function 2), turn the corresponding relay quickly **ON** and then **OFF**. The **ANALOG RANGE** display on the front panel will increase by one step. In a similar manner, Function 3 causes the **ANALOG RANGE** to decrease by one step.

3.7.2 PARALLEL REMOTE OPERATION

When using Parallel Remote, all detector functions are controlled remotely by computer through a computer interface. With the exception of **LOCAL/REMOTE, LIST,** and **DISPLAY** all front panel controls are disabled. Parallel Remote also allows remote monitoring of the detector output by the controller.

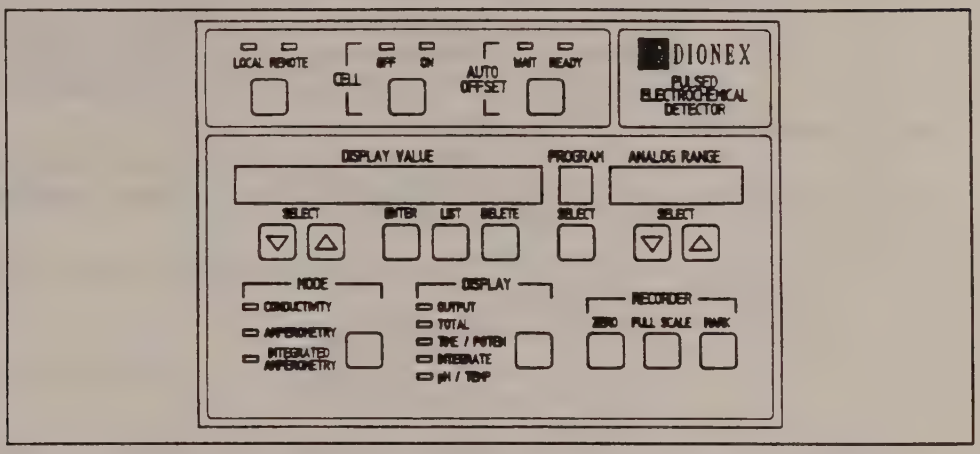
To select Parallel Remote operation, set the detector **LOCAL/REMOTE** switch to **REMOTE**. Consult the manual from you computer control system for complete instructions.

See Appendix C for 50-Pin Connector parallel I/O definitions and interface commands.

SECTION 4
CONTROLS, DISPLAYS, AND CONNECTORS

4.1 FRONT PANEL

Figure 4 - 1
Front Panel of the PED



- **LOCAL/REMOTE** allows selection of either local or remote control of the detector via the parallel interface. At power-up, the detector is in **LOCAL** operation. Pressing the switch selects **REMOTE** operation. If the detector is not connected to a parallel interface when **REMOTE** is selected, the **REMOTE** LED will flash continuously until the switch is reset to **LOCAL** or until the detector is connected to a controller. When the detector is used with Relay **REMOTE** operation, this switch must be in the **LOCAL** position.

- **CELL** controls the cell drive. Indicators above the **CELL OFF/ON** switch light to show the current state. Turning the cell drive **ON** applies voltage to the cell electrodes, enabling detection. Turning the cell drive **ON** also stores all programs in memory and implements any changes to the programs.

- **AUTO OFFSET** "zeros" the detector output. The microprocessor stores a measured value as an offset value the instant the switch is pressed. While **AUTO OFFSET** is **READY**, the output from the detector is the total measured value minus the offset value. The actual detector output level to the recorder following an **AUTO OFFSET** is selected by the user; settings of 0-70% of full scale, in 10% increments, are available. Most desktop integrators will not accept a negative signal. Setting the baseline above zero helps prevent a negative signal (due to negative baseline drift) from being sent to the integrator.

- **DISPLAY VALUE** provides an alphanumeric display for the currently selected **DISPLAY** function. Error messages also appear here.

- **PROGRAM** allows you to combine a set of parameters and store them as a program. Fifteen programs are stored in memory: five (A through E) for **AMPEROMETRY**, and ten (0 through 9) for **INTEGRATED AMPEROMETRY**. The detector cannot operate in **AMPEROMETRY** or **INTEGRATED AMPEROMETRY** modes unless it has been programmed to do so. **PROGRAMS** are not used in the **CONDUCTIVITY** mode.

- **PROGRAM SELECT** allows you to recall a program, either for review or to run.

- The **ANALOG RANGE** switches increase (via the **SELECT** arrow pointing up) or decrease (via the **SELECT** arrow pointing down) the full-scale range of the detector output to the recorder. The ranges are in siemens, amperes, or coulombs, depending on whether the selected detection mode is **CONDUCTIVITY**, **AMPEROMETRY**, or **INTEGRATED AMPEROMETRY**.

  When using Relay Control to control the detector, momentarily turning **ON** Function 2 causes the **ANALOG RANGE** to increase; Function 3 causes the **ANALOG RANGE** to decrease. The output range displayed on the front panel will correspond to the new setting.

- Press **ENTER** to accept the value currently displayed under **DISPLAY VALUE** and assign it to the parameter currently selected. Changes to programs are not implemented until the cell is turned **ON**.

- Press **LIST** to view each step, one-at-a-time, in a program. Steps appear in **DISPLAY VALUE**.

- **DELETE** has five functions:

  1. If you are changing a parameter, pressing **DELETE** once resets the parameter to the value last entered for it.
  2. If you are in the **TIME/POTEN** or **INTEGRATE** mode and are currently displaying a time other than 0.00 seconds, pressing **DELETE** immediately deletes the selected program step and displays the next step. If there is no next step, the step preceding the one just deleted will be displayed.
  3. If you are in the **TIME/POTEN** mode, and the display shows a time of 0.00 seconds, and the cell is **OFF**, pressing **DELETE** once causes the message **DELETE PGM?** to appear under **DISPLAY VALUE**. Press **ENTER** to delete the program. If you do not wish to erase the program, press any other button on the front panel to cancel the **DELETE** command.
  4. If you are in the **TIME/POTEN** mode, and the display shows a time of 0.00 seconds, and the cell is **ON**, pressing **DELETE** once causes the message **RESTORE PGM?** to appear under **DISPLAY VALUE**. Press **ENTER** to restore the currently-running program. If you do not want to restore the program, press any other button on the front panel to cancel the **RESTORE** command.
  5. If an error message appears in **DISPLAY VALUE**, press **DELETE** to clear the message so that you can correct the error.

- Press the **MODE** switch to select the appropriate detection mode: **CONDUCTIVITY**, **AMPEROMETRY**, or **INTEGRATED AMPEROMETRY**.

- Press the **DISPLAY** switch to select any of the five display functions: **OUTPUT**, **TOTAL**, **TIME/POTEN**, **INTEGRATE**, and **pH/TEMP**.

  Select **OUTPUT** to view the offset output, which is the actual analog output to the recorder. **OUTPUT** is displayed in siemens, amperes, or coulombs, depending on whether the selected detection mode is **CONDUCTIVITY**, **AMPEROMETRY**, or **INTEGRATED AMPEROMETRY**, respectively.

  Select **TOTAL** to view the total output unadjusted by **AUTO OFFSET**. **TOTAL** is displayed in siemens, amperes, or coulombs, depending on whether the selected detection mode is **CONDUCTIVITY**, **AMPEROMETRY**, or **INTEGRATED AMPEROMETRY**.

  Select **TIME/POTEN** to view and change programmed times and potentials.

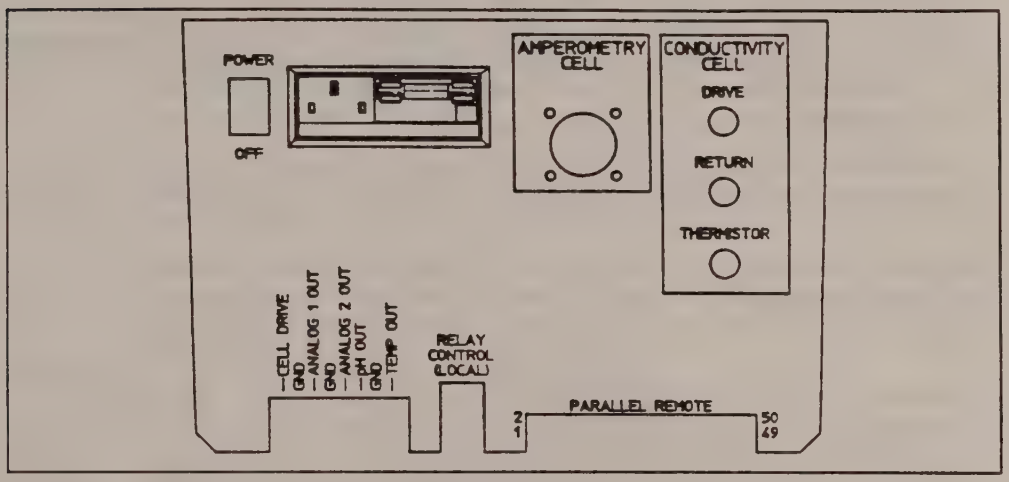
  Select **INTEGRATE** to choose the times at which integration begins and ends during integrated amperometry. A maximum of eight integration periods can be specified. The charge from each integration period is added to the total.

NOTE: **The total time allowed for integration is limited to between 80% and 90% of the total program time, depending on the program. An error message alerts you if the integration period is too long.**

Select **pH/TEMP** to display the pH of the mobile phase in the amperometry cell. In conductivity mode, both the temperature compensation factor and cell temperature are displayed.

- **ZERO** temporarily sets the detector output to the recorder to 0 V in order to zero the recorder. The display and the actual cell output are unaffected.
- **FULL SCALE** sends a full-scale signal to the recorder to adjust the recorder full-scale deflection. The display and the actual cell output are unaffected.
- **MARK** sends a positive pulse of 10% of full scale to the recorder as an event marker.

Figure 4 - 2
Rear Panel of the PED



4.2 REAR PANEL

- **POWER** controls the AC power to the detector.
- The Fuse/Voltage-Select unit contains the fuse, the voltage select card, a line filter, and the power cord connection. Operating voltages of 100, 120, 220, or 240 VAC can be selected.
- **AMPEROMETRY CELL** provides a connection to the amperometry cell.
- The **CONDUCTIVITY CELL DRIVE** and **CELL RETURN** connectors provide connections from the detector to the conductivity cell electrodes. The **CELL THERMISTOR** connector provides a connection from the conductivity cell thermistor to the detector. The cable labeled **T** from the conductivity cell is connected here.
- The **RELAY CONTROL** connector provides a connection to one or more contact-closure relays for Relay Control operation, using a suitably-equipped integrator or other control device.
- **Analog Outputs** (at the bottom left of the panel) may be connected to a strip chart recorder, an integrator, the analog input of a computer interface; or, for cyclic voltammetry, an X-Y recorder or oscilloscope. Use the red and black wire assembly to connect to the recording device. Two assemblies are supplied in the ship kit. Attach the wires to the analog output connectors by slipping the wires into the appropriate slots and tightening the screws. The black wire connects to an output labeled GND (ground), and the red (positive) wire connects to the appropriate analog output.

The analog outputs are listed below:

CELL DRIVE - This output provides the potential applied to the amperometry cell in the **AMPEROMETRY** and **INTEGRATED AMPEROMETRY** modes. It is most commonly used to drive the X axis of an X-Y recorder during cyclic voltammetry.

ANALOG 1 OUT - This is the recorder connection for the detector analog output.

ANALOG 2 OUT - This connection is for future options and is not used at this time.

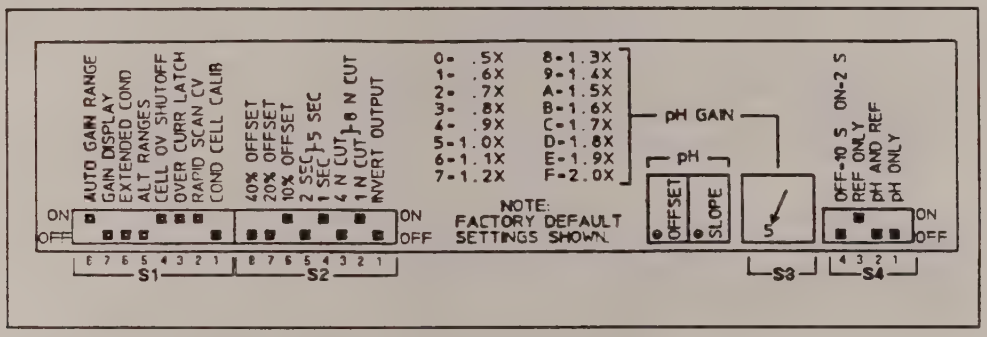
pH OUT - When the amperometry cell is being used with the combination pH-Ag/AgCl reference electrode, a voltage of 0.1 volts per pH unit is supplied at this output. Zero volts is pH 7. pH's above 7 provide positive voltages, pH's below 7 provide negative voltages.

TEMP OUT - In **CONDUCTIVITY** mode, a voltage of 0.1 volts per degree C is supplied at this output. Zero volts is 25 °C. Temperatures above 25 °C provide positive voltages; temperatures below 25 °C provide negative voltages.

4.3 PRINTED CIRCUIT BOARD SWITCHES

Three sets of printed circuit board switches (S1, S2, and S4) and one rotary switch (S3) are accessible through the top cover of the detector cabinet.

Figure 4 - 3
Printed Circuit Board Switches



4.3.1 Switch S1

Switch S1 is used to control miscellaneous functions.

- **Position 1: Conductivity cell calibration.** Default setting: **OFF.** When position 1 is in the **ON** position, cell calibration may be changed with the **SELECT** buttons on the front panel under the **DISPLAY VALUE.** Turn position 1 **OFF** to store the new calibration value. (Section 3.1.2).
- **Position 2: Rapid scan cyclic voltammetry.** Default setting: **ON.** The PED can perform two modes of cyclic voltammetry: Normal Scan (position 2 **OFF**), and Rapid Scan (position 2 **ON**). In Normal Scan, programs up to 99.9 s can be used. Data is collected at a rate of 20 measurements per second, or one measurement every 50 ms. In Rapid Scan (position 2 **ON**), data is collected at a rate of 100 measurements per second, or one measurement every 10 ms. Total program length is limited to 9.99 s. When position 2 is **ON**, the times for programs previously written in Normal Scan are divided by 10. Conversely, when position 2 is turned **OFF**, all times are multiplied by 10. (Section 3.3.2).

- **Position 3: Overcurrent latch.** Default setting: **ON**. If the cell current exceeds the maximum measurable amount, the warning message OVER CURRENT appears. When position 3 is **ON**, this message is not removed until **DELETE** is pressed. This is to inform you of the overcurrent condition if it occurs while you are not looking at the front panel. If an overcurrent condition is a routine but transitory occurrence, turning position 3 **OFF** causes the message to be deleted automatically when the cell current drops below the maximum measurable amount. Typical situations where it may be preferable to turn position 3 **OFF** are in cyclic voltammetry if the current temporarily goes beyond the maximum measurable amount at the potential limits; or in DC, integrated, or pulsed amperometry when one peak (for example a void volume upset) produces a very high current.

- **Position 4: Cell overvoltage shutoff.** Default setting: **ON**. If the cell is **ON** while the reference electrode is disconnected (either because it is physically disconnected or because the reference compartment is dry), the PED applies its maximum voltage between the working electrode and the counterelectrode. When this value is reached, the warning message OVER VOLTAGE is displayed. When position 4 is **ON**, the cell is also turned **OFF** if the voltage reaches the maximum. This is to protect the working electrode from damage.

- **Position 5: Alternate cell ranges.** Default setting: **OFF**. In the **OFF** position, the output ranges are in increments of 1, 3, 10, 30, etc. In the **ON** position, the output ranges are 1, 2, 5, 10, 20, 50, etc.

- **Position 6: Extended conductivity.** Default setting: **OFF**. Position 6 **OFF** allows conductivities of up to 750 µS to be measured. With position 6 **ON**, conductivities up to 1500 µS can be measured, however, the noise level is about twice as high as in the **OFF** position.

- **Position 7: Gain display.** Default setting: **OFF**. The PED uses eight internal levels of amplification (gain) which are automatically switched depending on the cell current. Since it is not necessary to know what the gain setting is, the level is normally not displayed on the front panel. Turning position 7 **ON** displays the gain setting on the front panel. This is only useful during instrument troubleshooting.

- **Position 8: Auto gain ranging.** Default setting: **ON**. Setting position 8 **OFF**, with Position 7 **ON**, allows manual setting of the internal gain (see explanation of position 7). Use the **SELECT** scroll buttons (located on the front panel under **DISPLAY VALUE**) to set the gain. Turning 8 to the **ON** position causes the PED to set the gain automatically.

4.3.2 Switch S2

Switch S2 is used to control output direction, filter settings and offset settings.

- **Position 1: Invert output.** Default setting: **OFF**. Turning **ON** position 1 causes negative response to be displayed as a positive output. (Response is either conductivity or current in the **AMPEROMETRY MODE**, or charge in the **INTEGRATED AMPEROMETRY MODE**.) The front panel display, baseline offset, full scale, and mark are unaffected and are always positive. This option is useful for displaying peaks instead of dips when performing indirect conductimetric detection, or for amperometry or integrated amperometry of reduction reactions.

- **Positions 2 and 3: 1-, 4-, and 8-nano cutoff.** These switches disable the filter on the output while a peak is being recorded. The purpose is to minimize peak distortion caused by filtering, and to increase accuracy. The combined use of switch positions 2 and 3 cause the filter to be disabled when the output between events changes by more than the values shown in Figure 4-4.

Figure 4 - 4
1-,4-, and 8-Nano Cutoff of Output Filter

SWITCH S2 POSITIONS 2	3	OUTPUT CHANGE REQUIRED TO DISABLE TIME CONSTANT FILTER
ON	OFF (Default)	1 nS, 1 nA, 1 nC
OFF	ON	4 nS, 4 nA, 4 nC
ON	ON	8 nS, 8 nA, 8 nC
OFF	OFF	Cutoff Disabled

- **Positions 4 and 5: 1-, 2-, and 5-second time constants on output filter.** These switch positions enable time constant filters whose purpose is to reduce short-term noise. The combined use of positions 4 and 5 select the time constant values shown in Figure 4-5.

Figure 4 - 5
1-, 2- and 5-Second Time Constants of Output Filter

SWITCH S2 POSITIONS 4	5	TIME CONSTANTS
ON	OFF (Default)	1 s
OFF	ON	2 s
ON	ON	5 s
OFF	OFF	Filter Disabled

- **Position 6, 7, and 8: 10% - 70% offset.** These switch positions are used to set the baseline to a selected percentage of full scale when **AUTO OFFSET** is pressed. This is useful to prevent a small drift in the baseline from going to zero. The PED cannot provide a negative output, and most integrators and computer data systems cannot record negative outputs. The combined use of switch positions 6, 7, and 8 provide offsets from 10% to 70% of full scale in 10% increments as show in Figure 4-6.

Figure 4 - 6
Baseline Offset Percentage

SWITCH S2 POSITIONS 6	7	8	OFFSET PERCENTAGE
ON	OFF	OFF (Default)	10%
OFF	ON	OFF	20%
ON	ON	OFF	30%
OFF	OFF	ON	40%
ON	OFF	ON	50%
OFF	ON	ON	60%
ON	ON	ON	70%
OFF	OFF	OFF	Offset Disabled

4.3.3 Switch S3

This switch controls pH gain. It is a 16-position rotary switch which allows the voltage response vs. pH of the pH reference electrode to be multiplied by factors of 0.5 to 2. This is useful for correcting alkaline error: a decrease in the volts per pH unit response of glass electrodes at high pH. This correction applies only when position 2 of S4 is in the pH and Ref. position (ON). Only the potentials applied to the cell are corrected; the front panel pH read-out and the rear panel pH analog output are unaffected. The switch positions and corresponding gains are listed on the label on the top cover. See Section 3.5.2 for an explanation of how to adjust the applied potentials when the pH reference electrode is used with this correction.

4.3.4 Switch S4

Switch S4 is used for reference electrode selection.

- **Position 1: pH only.** Default setting: **OFF**. When position 1 is **ON**, the amperometry cell reference potential is controlled by the pH half cell of the combined pH-Ag/AgCl reference electrode. (See Section 3.5.) When position 1 is **ON**, positions 2 and 3 must be **OFF**.
- **Position 2: pH and Reference.** Default setting: **OFF**. When position 2 is **ON**, the amperometry cell reference potential is controlled by the pH half cell of the combination pH-Ag/AgCl reference electrode. However, the Ag/AgCl half cell provides a reference to the pH half cell. This decreases baseline noise as compared to the pH half cell only, and allows the voltage response of the pH electrode to be multiplied by the gain set by the 16-position rotary switch, S3 (see section 3.5). When position 2 is **ON**, positions 1 and 2 must be **OFF**.
- **Position 3: Reference only.** Default setting: **ON**. When position 3 is **ON**, the amperometry cell reference potential is controlled by the Ag/AgCl half cell of the combination pH-Ag/AgCl reference electrode, or by the single Ag/AgCl reference electrode if it is being used. When position 3 is **ON**, positions 1 and 2 must be **OFF**.
- **Position 4: Response time for pH reference.** Default setting: **OFF**. This position controls the response time of the pH reference electrode to changes in pH when S4 is set to position 2. When position 4 is **OFF**, the response time is 10 s. When position 4 is **ON**, the response of the electrode to mobile phase pH changes is faster (2 s), but the baseline may be slightly noisier.

4.4 ERROR AND WARNING MESSAGES

Following is an alphabetical listing of error messages that can appear while programming or operating the detector.

Error Messages indicate a condition which prevents the detector from operating. When an error message appears, you must first clear the message by pressing **DELETE**, then follow the directions found in the listing below to correct the condition which caused the error message. Finally, turn the cell **ON** again.

Warning Messages indicate that a non-standard practice has been performed by the operator. This does not necessarily mean that the procedure is incorrect. The message is merely a warning.

MESSAGE	POSSIBLE CAUSE
CELL ERROR	1. Detector cell error condition. Press **DELETE** to view next message. 2. Detector cell is not connected. Make sure the cell is correctly installed (see Section 2, Installation). 3. Failure of analog board circuitry. Contact your nearest Dionex Service Office (see Section 2.1).
DELETE PROGRAM	If the time is set at time 0.00 seconds and the cell is **OFF**, pressing **DELETE** once causes the **DELETE PROGRAM?** message to appear. If you wish to delete the program, press **ENTER**. Delete the command by pressing any other button on the front panel.
DMA ERROR	Cycling of waveform on the analog board timed out. The analog board may be defective. Contact the nearest Dionex Service Office (see Section 2.1).

dV <> INTEGER	A cyclic voltammetry waveform has been programmed that results in uneven ramp steps. This may cause oscillations in baseline. (See Section 3.3.3.)
END = BEGIN	You tried to enter an integration window with an end time less than or equal to the begin time. Re-enter the time.
END <> BEGIN	A cyclic voltammetry wave form has been programmed with an ending potential not equal to the beginning potential. This is a warning only. Press **DELETE** to clear message, and proceed with normal operation.
FULL ERROR	1. All available program memory has been used. To create more memory, delete an unused program. 2. Possible failure of the computer board. Contact your Dionex Service Office (see Section 2.1).
INTEG. >8	The maximum number of integration windows has been exceeded. Edit program to reduce the number of windows to eight or fewer.
INTEG. OVERLAP	Two integration windows with overlapping times have been entered. Change program so that integration windows do not overlap
INTEG. > X.XX s	Integration window is longer than that allowed for the program. The displayed value is the maximum integration time allowed for the current program.
NEW NVRAM	RAM memory problem. Contact your Dionex Service Office (see Section 2.1).
NO NVRAM	RAM memory problem. Contact your Dionex Service Office (see Section 2.1).
NO ANALOG BD	Indicates that no analog board is installed. Contact the nearest Dionex Service Office (see Section 2.1).
NO INTEG.	Indicates that no integration period is programmed in integrated amperometry. Enter integration period. (See Section 3.4.2.)
NO OPTION BD	Indicates that an optional second working electrode board is not installed.
NO TIME/POT	No waveform was entered for this program. Enter a waveform for integrated amperometry. (See Section 3.4.2.)
NV RAM ERROR	Indicates an error in writing or reading memory. Contact the nearest Dionex Service Office (see Section 2.1).
OVER CURRENT	Maximum allowable current has been exceeded. Modify potentials in program to reduce peak currents. (See Section 4.3.1, Position 3, or Section 5.8.)
OVER VOLTAGE	Potential applied to cell has exceeded safe operating levels. 1. Reference electrode is not connected (see Section 2.3.4.2). 2. Detector cable is not connected or is broken. 3. Failure of analog board (contact you Dionex Service Office (see Section 2.1).
PGM ERROR	Invalid selection in program. Press **DELETE** to view the next message.

PGM WARNING	Non-standard (but not fatal) operation selected. Press **DELETE** and proceed.
RAM FAILURE	Power up diagnostics has found a problem with with random access memory. Contact the Dionex Service Office (see Section 2.1).
RESTORE PGM	If the time is set at 0.00 seconds and the cell is **ON**, pressing **DELETE** once causes the **RESTORE PGM** message to appear. If you want to restore the program, press **ENTER**. Cancel the command by pressing any other button on the front panel.
TEMP. FAIL!	Indicates failure of autocalibration of temperature measurement circuits, caused by a defective analog board. Contact your Dionex Service Office (see Section 2.1).
TIME < 0.10 s	For integrated amperometry, the minimum waveform cycle time is 0.10 s. Re-enter the cycle time.

SECTION 5
TROUBLE-SHOOTING

This section is designed to help isolate and solve problems. To solve a problem, first select the symptom description that best describes your problem. (Symptom descriptions are numbered 5.1 through 5.12 and printed in bold capital letters.) After you select a symptom, consider the **Possible Cause(s)** shown, and then take the action recommended in the **Possible Remedy** column. If the problem persists, call the Dionex Service Office nearest you (see Section 2.1).

5.1 NO DETECTOR RESPONSE

POSSIBLE CAUSE	POSSIBLE REMEDY
A. The **CELL** drive indicator is **OFF**.	Press the **CELL** front panel switch to turn the drive **ON**.
B. The ANALOG RANGE to recorder is set too high. The display indicates a response, but no recorder response is observed.	Select a more sensitive **ANALOG RANGE**.
C. A cell cable is not connected.	1. Make sure that the cell cables are connected. 2. Reset the detection process by pressing the **CELL** switch twice to turn the **CELL OFF**, then turn it **ON** again.
D. You select **REMOTE** operation; the **REMOTE** LED flashes continuously.	Check that the detector is properly connected to an interface. If the interface 50-pin parallel cable is not properly seated in the parallel connector on the detector rear panel, or if the interface is not turned on, the front panel **REMOTE** led will blink continuously. NOTE: For manual control of the detector, press the **LOCAL/REMOTE** switch to select **LOCAL** mode.
E. No Full-Scale Output selected on the high-resolution output board.	Select 0.01, 0.10, or 1 V Full Scale on the high-resolution output board.
F. Pump is not pumping.	Check the pressure reading on the pump to ensure that the pump is on.
G. Detector offset out of range.	Press **AUTO OFFSET**.
H. Incorrect connections to **ANALOG 1 OUT** on rear panel.	Ensure that the recorder is properly connected to **ANALOG 1 OUT** on the rear panel.

5.2 LOW DETECTOR OUTPUT

POSSIBLE CAUSE	POSSIBLE REMEDY
A. The **ANALOG RANGE** to recorder is set too high. The display indicates a response, but little or no recorder response is observed.	Select a more sensitive **ANALOG RANGE**.
B. An insufficient sample is injected.	Increase the injection size or concentration.
C. Conductivity cell is out of calibration.	Recalibrate cell (See Section 3.1.2).
D. Working electrode is fouled.	Polish working electrode (See Section 2.3.4.1).

5.3 HIGH DETECTOR OUTPUT

POSSIBLE CAUSE	POSSIBLE REMEDY
A. **AUTO OFFSET** has not been activated recently.	1. Press **AUTO OFFSET** before making an injection. 2. When using Relay Control, offset the background by pulsing on the relay controlling the **AUTO OFFSET** function.
B. In **CONDUCTIVITY MODE**, the regenerant is not suppressing the background.	Use a higher regenerant flow rate.
C. In **INTEGRATED AMPEROMETRY MODE**, too many integration periods. Integration periods not properly deleted during editing.	Set **DISPLAY** to **INTEGRATE, LIST** Integration periods. Delete unnecessary periods.

5.4 NOISY BASELINE

POSSIBLE CAUSE	POSSIBLE REMEDY
A. Air trapped in the cell; regular pulses in baseline.	Remove trapped air. Increase the length of the wasteline to increase the backpressure on the cell.
B. There is a flow system leak ahead of the cell; the baseline is erratic.	Check all fittings and liquid lines for leaks. Tighten any leaking fittings finger-tight, then tighten an additional 1/8 turn. Tighten further only if the leak continues. **DO NOT OVER-TIGHTEN!** If tightening does not stop the leak, remake the fitting.
C. In **CONDUCTIVITY MODE**, operating conditions for the Membrane Suppressor are inappropriate.	Operate the suppressor as directed in the suppressor manual.
D. Pump not properly primed.	Reprime pump.
E. In DC Amperometry Mode, there is rapid and large oscillation in the baseline, caused by more than one potential.	Set **DISPLAY** to **TIME/POTEN, LIST** Program; delete unnecessary potentials.
F. In **AMPEROMETRY** and **INTEGRATED AMPEROMETRY** Modes, there are frequent, random spikes in the baseline, caused by a plugged reference electrode.	Replace reference electrode.
G. In **AMPEROMETRY** and **INTEGRATED AMPEROMETRY MODES**, there is regular baseline oscillation on high-sensitivity ranges, caused by not using the short stainless-steel tubing on the cell inlet.	Reconnect tubing to cell inlet.
H. In **AMPEROMETRY** and **INTEGRATED AMPEROMETRY**, the working electrode is dirty.	Polish the working electrode . (see Section 2.3.4.1).
I. There are rapid temperature changes in the laboratory. A heater or air-conditioner is blowing directly on the chromatograph.	Direct heating and air-conditioning vents away from the chromatograph.

5.5 DRIFTING BASELINE.

POSSIBLE CAUSE	POSSIBLE REMEDY
A. Insufficient system equilibration following any changes to the operating parameters; especially apparent when operating at high sensitivities.	Allow longer system equilibration before beginning operation.
B. In **CONDUCTIVITY MODE**, the cell - THERMISTOR cable is not attached to the **CELL THERMISTOR** connector on the rear panel.	1. Connect the cell thermistor cable to the **CELL THERMISTOR** connector. 2. Reset the detection process by turning **CELL OFF** and then back **ON**.
C. In **CONDUCTIVITY MODE**, the **TEMPERATURE COMPENSATION** setting is unoptimized.	Optimize the selected setting (see Section 3.1.1).
D. In **CONDUCTIVITY MODE**, the operating conditions for the Membrane Suppressor are inappropriate.	Operate the suppressor as instructed in the suppressor manual.
E. There is a flow system leak ahead of the cell; the baseline is erratic.	Check all fittings and liquid lines for leaks. Tighten any leaking fittings finger-tight, then tighten an additional 1/8 turn. Tighten further only if the leak continues. DO NOT OVER-TIGHTEN! If tightening does not stop the leak, remake the fitting.
F. In **AMPEROMETRY** and **INTEGRATED AMPEROMETRY** modes, the working electrode is dirty.	Polish the working electrode (See Section 2.3.4.1).
G. There are rapid temperature changes in the laboratory. A heater or air-conditioner is blowing directly on the chromatograph.	Direct heating and air-conditioning vents away from the chromatograph.

5.6 FLASHING REMOTE LED

POSSIBLE CAUSE	POSSIBLE REMEDY
REMOTE operation is selected, and there is no connection to the computer interface.	Check that the detector is properly connected to a computer interface. If the Controller 50-pin parallel cable is not properly seated in the parallel connector on the detector rear panel, or if the interface is not turned on, the front panel **REMOTE LED** will blink continuously. NOTE: For manual control of the detector, press the **LOCAL/REMOTE** switch to select **LOCAL** Mode.

5.7 FLASHING PROGRAM NUMBER

POSSIBLE CAUSE	POSSIBLE REMEDY
The program currently running was edited, or a program other than the one which is running was selected.	Press **PGM** to revert to the currently running program, or turn the cell **OFF** and then back **ON** to implement the flashing program.

5.8 CELL OVERCURRENT ERROR MESSAGE

POSSIBLE CAUSE	POSSIBLE REMEDY
A. In **INTEGRATED AMPEROMETRY MODE**, a program's integration period ramp or step is too large.	1. Increase ramp time during the integration period. For example, change a ramp from 0 V to +0.8 V in 0.05 s to 0.10 s or 0.20 s. 2. Decrease the total voltage ramp during integration periods; avoid potential limits.
B. Too many integration periods.	Set **DISPLAY** to **INTEGRATE**. **LIST** integration periods; delete unnecessary periods.

5.9 CELL OVERVOLTAGE ERROR MESSAGE

POSSIBLE CAUSE	POSSIBLE REMEDY
Reference electrode is not connected.	1. Check all connectors to the reference electrode. 2. Ensure that the cell is not dry and that the reference compartment is filled with mobile phase. 3. If reference electrode is plugged, replace it.

5.10 pH READOUT FROM AMPEROMETRY CELL ALWAYS 7.0

POSSIBLE CAUSE	POSSIBLE REMEDY
Short circuit in pH reference electrode	1. Check connections to cell preamp PC-board. 2. Replace pH electrode.

5.11 NO pH READOUT FROM AMPEROMETRY CELL

POSSIBLE CAUSE	POSSIBLE REMEDY
A. Ag/AgCl only reference electrode being used.	Replace with combination pH-Ag/AgCl reference electrode.
B. One or two pH electrode wires disconnected or broken.	Fix connections or replace electrode.
C. pH electrode not calibrated.	Calibrate pH electrode (see Section 2.3.4.3).

5.12 pH READOUT FROM AMPEROMETRY CELL CAN NOT BE SET TO 7.0. TURNING OFFSET POTENTIOMETER CAUSES DISPLAY TO CHANGE.

POSSIBLE CAUSE	POSSIBLE REMEDY
A. Calibration buffer not accurate.	Use a separate pH meter to check pH of buffer.
B. pH reference electrode contaminated.	1. Soak electrode in a 1 M KCl, 1 M HCl solution. 2. Replace electrode.

APPENDIX A
PED SPECIFICATIONS

Modes
Conductivity
DC Amperometry and Cyclic Voltammetry
Integrated & Pulsed Amperometry

Potential vs. Time Waveforms

Maximum number of potentials in program: 100
Potential ranges: ± 2.00 V in 0.01 V increments

Times:
Cyclic Voltammetry: 0.00 to 9.99 s in 0.01 s increments (Rapid Scan)
00.0 to 99.9 s in 0.01 s increments (Normal Scan)
Integrated & Pulsed Amperometry: 0.00 to 2.00 s in 0.01 s increments

Front Panel Digital Readouts

Autoranging conductivity, current, or charge. Total or after offset
Potential vs. time waveform programs
Integrated amperometry integration periods
Amperometry cell pH
Conductivity cell temperature
Conductivity cell temperature compensation factor

Rear Panel Outputs

Digital parallel to computer interface
Analog to integrator or recorder
Amperometry cell pH: 0.1 V/pH unit (pH 7 = 0.0 V)
Conductivity cell temperature: 0.1V/ C (25° C = 0.0 V)
Applied Potential (X axis for cyclic voltammetry)

Filter:
1, 2, & 5 s time constants

Ranges:

1 nanounits to 5 milliunits in steps of 1, 2, 5, 10; or 1, 3, 10 (DIP switch selectable)

Units:
Conductivity: S (siemens)
Amperometry: A (amperes)
Integrated Amperometry: C (coulombs)

Offset:
Auto Offset to 1.25mS, 1.25 mA, or 1.5 mC

Full scale analog output:
0.01, 0.1, or 1 V
Baseline position:
0 to 70% of full scale in 10% increments

Conductivity

Cell volume: 1.5 µL
Max. cell pressure: 20 bar (300 psi)
Noise: approximately 500 pS (with TS-1 option)
Temperature compensation: 0 to 9.9%/C

Amperometry

Cell type: Thin layer, reference electrode in Faraday cage
Cell materials: 316 stainless-steel and Kel-F
Thin layer channel volume: 3.5 µL
Thin layer channel thickness: 76 µm (0.003")
Max. cell pressure: 7 bar (100 psi)
Working electrodes: Gold, Platinum, Silver, or Glassy Carbon
Working electrode diameter: 3 mm
Reference electrodes: pH-Ag/AgCl combination or optional Ag/AgCl
DC Amperometry noise: Approximately 30 pA (GC electrode)
Integrated Amperometry noise: Approximately 60 pC (Au electrode)
Potential vs. time programs stored in memory:
DC Amperometry & Cyclic Voltammetry: 5
Integrated & Pulsed Amperometry: 10
Max. CV scan rate: Dependent on potential limits and desired resolution.
5 V/s for 0 and 1 V limits and 0.05 V resolution.
(Allows "on-the-fly" CV. Requires X-Y plotter or storage oscilloscope)

Power requirements: 100, 120, 220, 240 V AC, 50/60 Hz, 60 W

Temperature requirements: 10° to 40° C (50° to 104° F)

Dimensions: (H x W x D): 16.5 x 22 x 42 cm (6.5" x 8.5" x 16.5")

Weight: 4.5 Kg (10 lbs.)

APPENDIX B
RELAY REMOTE CONNECTIONS

Relay Control uses a six-pin telephone-style connector. Each Relay Control function is operated by a separate contact-closure relay or TTL logic level. When the connector key is up, the connector pins are numbered from right to left, with pin 1 on the right and pin 6 on the left. Table 1 lists the six pins and the functions they control. Line 6 (ground) is required for each function.

When the function is inactive, the relay contacts are open and the lines are *pulled* high to a +5 volts. Turning the function on closes the relay contacts, grounding the corresponding line and dropping it to zero volts (lo).

PIN	COLOR	NAME	FUNCTION
1	Orange	+5 V DC	Supplies power to an external load (50 µA, maximum).
2	Black	AUTO OFFSET	Function 1: Detector Auto Offset Relay off (hi): No Effect Relay on (lo): Zero detector output *
3	Red	RANGE UP	Function 2: Increase RANGE Relay off (hi): No Effect Relay on (lo): Output range from detector is incremented 1 step; front panel is updated accordingly.
4	Green	RANGE DOWN	Function 3: Decrease RANGE Relay off (hi): No Effect Relay on (lo): Output range from detector is decremented 1 step; front panel is updated accordingly.
5	Yellow	REMOTE	The controller must be grounded. If it is not grounded, the detector will not respond to Relay Control commands.
6	Brown	Ground	Supplied by the detector. Must be connected.

* Subtracts the background from subsequent measurements.

APPENDIX C
PARALLEL I/O DEFINITIONS AND INTERFACE COMMANDS

Pin No.	Port No.	Data Bit	Function
1	2	7	MON - Detector Type (1 = PED)
2	2	6	MON - Error
3	2	5	MON - Data Exponent - 16
4	2	4	MON - Data Exponent - 8
5	2	3	MON - Data Exponent - 4
6	2	2	MON - Data Exponent - 2
7	2	1	MON - Data Exponent - 1
8	2	0	MON - Data Ready
11	1	7	MON - Data -Sign (0 = positive, 1 = negative)
12	1	6	MON - Data - 16384
13	1	5	MON - Data - 8192
14	1	4	MON - Data - 4096
15	1	3	MON - Data - 2048
16	1	2	MON - Data - 1024
17	1	1	MON - Data - 512
18	1	0	MON - Data - 256
21	0	7	MON - Data - 128
22	0	6	MON - Data - 64
23	0	5	MON - Data - 32
24	0	4	MON - Data - 16
25	0	3	MON - Data -8
26	0	2	MON - Data - 4
27	0	1	MON - Data - 2
28	0	0	MON - Data - 1
29	0	N/A	MON - Data - Ready (Not Required)
30	Spare	N/A	
31			No Module (Module must ground
32			No Remote Control (Control must ground)
33	1	7	CMD - Load Strobe
34	1	6	CMD - Up/Down Load
35	1	5	CMD - Load/Run
36	1	4	CMD - Sample Stream A/B
37	1	3	CMD - Load Command - 8
38	1	2	CMD - Load Command - 4
39	1	1	CMD - Load Command - 2
40	1	0	CMD - Load Command - 1

(Continued on next page)

NOTES:

1. Commands with slashes refer to functions that are affected when line is at ground or at five volts (i.e., Cell Off/On indicates Off when line is high, and On when line is low).

2. Command port 0 is a direct command port.

3. Command port 1 is used to set modes and strobe commands for transferring programs to and from the PED.

Pin No.	Port No.	Data Bit	Function
41	0	7	CMD - Marker
42	0	6	CMD - Auto Offset
43	0	5	Display Total/Offset Output
44	0	4	CMD - Cell Off/On
45	0	3	CMD - Analog Range - 8 (100 pico, 300 pico,..., 1 milli, 3 milli)
46	0	2	CMD - Analog Range - 4
47	0	1	CMD - Analog Range - 2
48	0	0	CMD - Analog Range - 1
49	GROUND		
50	GROUND		

MP MASTER
MA32859